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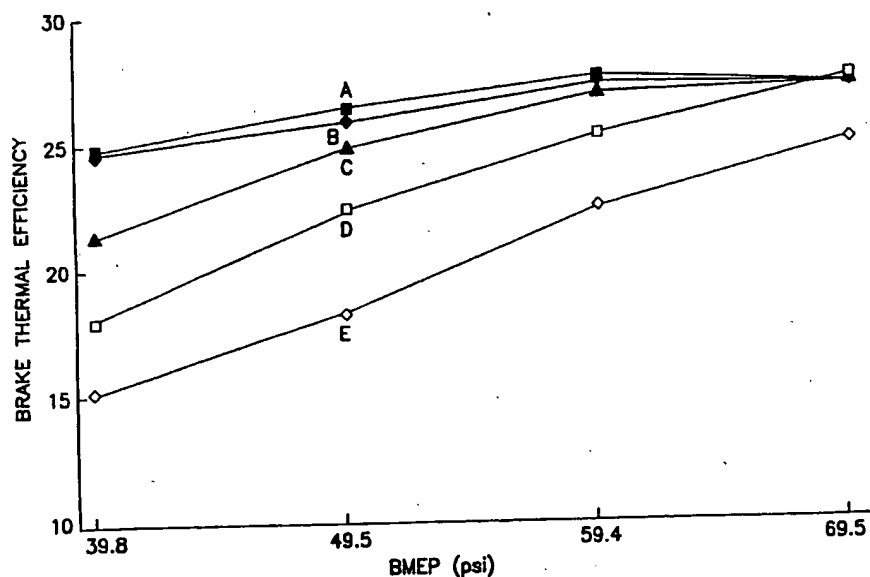
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(21) International Application Number: PCT/US93/05103 (22) International Filing Date: 28 May 1993 (28.05.93) (30) Priority data: 07/892,160 2 June 1992 (02.06.92) US (71) Applicant: GREENBRANCH ENTERPRISES, INC. [US/US]; 3 Crafts Road, Gloucester, MA 01930 (US). (72) Inventors: EARLE, Roland, D. ; 9241 West Broward Boulevard, #3512, Plantation, FL 33324 (US). HOL- LAND, Thomas, G. ; 11313 Boca Woods Lane, Boca Raton, FL 33428 (US). (74) Agent: LORUSSO, Anthony M.; Lorusso & Loud, 440 Commercial Street, Boston, MA 02109 (US).		(81) Designated States: AT, AU, BB, BG, BR, CA, CH, DE, DK, ES, FI, GB, HU, JP, KP, KR, LK, LU, MG, MN, MW, NL, NO, PL, RO, RU, SD, SE, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG). Published With international search report.

(54) Title: A PHASE STABILIZED ALCOHOL BASED DIESEL FUEL CONTAINING IGNITION ADDITIVES



(57) Abstract

A diesel fuel containing 10 % by volume ethanol (B), 20 % by volume ethanol (D), 25 % by volume ethanol (C) or 30 % by volume ethanol for a diesel was compared to the optimum brake thermal efficiency for 100 % diesel fuel (A) wherein the (B) diesel fuel composition has almost the same diesel characteristics of conventional diesel fuel (A). A method for powering a diesel internal combustion engine with fuel having as its primary components ethanol, diesel fuel, butanol and an alkyl peroxide.

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1.

BACKGROUND OF THE INVENTION

"A Phase Stabilized Alcohol Based Diesel Fuel Containing Ignition Additives"

This invention is a fuel for an internal combustion engine of the diesel type wherein ignition of the fuel is effected by the heat of compression of air in the cylinders and is more particularly concerned with producing a fuel composition containing alcohol which can replace the petroleum based diesel fuels now used as the fuel in such engines.

With the world's supply of petroleum diminishing, much emphasis has been placed on finding substitutes for fuels such as gasoline or diesel fuel which are derived from petroleum. This search has extended to fuels for all types of internal combustion engines, including those using gasoline and diesel fuel. For example, a combination of gasoline and ethyl alcohol sometimes called "gasohol" has been widely heralded because it lowers the amount of gasoline required to run an internal combustion engine. Of course, gasohol contains a large amount of gasoline (90%) and thus only slightly reduces dependence on petroleum products.

Most importantly, however, is that until the inventions set forth by Roland Earle in U.S. Patent No. 4,298,351, issued November 3, 1981, and U.S. Patent No. 4,386,938, issued June 7, 1983, the inclusion of alcohol in fuel blends was considered undesirable because alcohols such as ethanol were thought to be incapable of autoigniting and their inclusion in fuel in even minor amounts rendered the fuel less desirable in an internal combustion engine. Autoigniting is a phenomenon which can be described as follows. Generally, a diesel engine operates on the Carnot cycle. The cylinder, after being filled with air, is closed and a piston moves to compress the air. As the fuel enters the cylinder, with its high pressure and high temperature air

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environment, the fuel is atomized into small droplets, and vaporizes and mixes with the air. As long as the compressed air is capable of heating the fuel above the ignition temperature and pressure of the fuel, portions of the fuel will begin to autoignite. Until recently, the only truly acceptable fuel for such engines has been derived entirely from petroleum fuels. However, as is stated above, the Earle patents set forth an alcohol containing fuel that was found to be effective. The "Earle" fuel is based on the discovery that an organic peroxide in the fuel blend enables an alcohol to be included in a fuel in significant amounts and yield a fuel which will autoignite.

The use of peroxides as additives for petroleum-based fuels had been suggested prior to the Earle invention. For example, U.S. Patent No. 1,766,501 to Buerk entitled "Liquid Combustible" discloses adding peroxides in general to improve the combustion effect of gasoline.

U.S. Patent No. 3,108,864 to Barusch entitled "Engine Starting Fluid" describes the mixture of large quantities of dimethyl peroxide with diethyl ether as a starting primer for gasoline engines under sub-freezing conditions.

U.S. Patent Nos. 2,011,297 to Moser entitled "Process for Preparing Motor Fuel"; 2,092,322 to Moser entitled "Process for the Production of Organic Peroxides"; 2,093,008 to Egerton entitled "Fuel for Internal Combustion Engines"; 2,107,059 to Moser entitled "Motor Fuel Composition"; 2,174,680 to Badertscher et al. entitled "Diesel Fuel"; 2,240,145 to Moser entitled "Motor Fuel Composition" and 2,891,851 to Bailey et al. entitled "Fuel for Internal Combustion Engines" disclose the use of peroxides as additives to diesel fuel.

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U.S. Patent No. 2,696,806 to Mingle, Jr. entitled "Removal of Combustion Chamber Deposits in Spark-Ignition Engines" discloses adding a peroxide to a fuel for removing deposits in spark-ignition engines.

U.S. Patent No. 4,298,351 issued to Earle discloses a methanol based fuel which is improved by an alkyl peroxide additive. U.S. Patent No. 4,386,938 issued to Earle discloses a methanol or ethanol (and other lower alcohol) based fuel which is improved by an alkyl peroxide additive for use as a substitute for gasoline. However, these patents do not disclose the use of alcohol as a fuel in a diesel engine specially as the present invention teaches. Furthermore, no one has successfully used organic peroxides to enable alcohol to function in a diesel engine until the present invention set forth by Roland Earle. The preferred organic peroxide for inclusion in the "Earle" fuel is an alkyl peroxide and in particular is a di-tertiary alkyl peroxide. The most preferred additive is a di-tertiary butyl peroxide.

The invention set forth by Roland Earle for the inclusion of the organic peroxide additives enabling an alcohol to function in a diesel engine, also promotes co-solvency of alcohols, such as ethanol, and diesel which was previously difficult to accomplish. Such disparate fuels tend to separate under humid or cool conditions without such additives.

Even with the discoveries noted in the Earle patents for alcohol based fuels, however, an additional problem remains. Water absorption in alcohol based diesel fuel has been an additional concern in the past. If even a small quantity of water gets into the fuel, an alcohol based diesel fuel will separate. Thus, it would be most beneficial to have an alcohol based diesel fuel which does

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not phase separate with the absorption of water. Phase stability, as described herein, refers to the ability of the fuel to refrain from separating into its component phases. Phase separation problems as encountered with water content are also sensitive to temperature. The problems of autoignition and phase stability, associated with the inclusion of an alcohol to a diesel fuel entail exceptional costs, it would be desirable to address these concerns in a cost effective way.

The use of a clean-burning alcohol fuel to replace diesel is desirable to address the urgent problem of environmental pollution, and in particular to reduce major pollutants in emissions such as NO_x , CO, CO_2 , sulphur and particulate matter. An alcohol containing fuel could be made available to the heavy duty diesel fuel industry in particular, vehicles such as municipal sanitation trucks, buses, Post Office and parcel delivery trucks. All that would be required for implementation of the fuel of the present invention in, for example a fleet of municipal sanitation trucks, would be to install mixing equipment in the fleet refueling station. In addition, ethanol can be produced simply from domestically grown feedstock, decreasing the dependency on largely imported diesel. Further, recent technological advances in the production of ethanol will cut the cost of its production and thereby make an alcohol based fuel a highly desirable commodity.

Thus, it would be desirable to provide an alcohol based diesel fuel composition which addresses the concerns of autoignition and phase stability, and which can be manufactured in a cost effective way; to produce a fuel which performs with the same characteristics as straight diesel fuel, and also significantly reduce emissions.

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SUMMARY OF THE INVENTION

In accordance with the present invention, it has been discovered that a fuel of the following composition can enable an alcohol to be included in a diesel fuel: an alcohol/diesel oil blend of approximately 30/70% by volume; a phase stability additive selected from propanol, butanol, hexanol or dodecanol in an amount between approximately 4.5 and 5.5% by volume; an autoignition and co-solvency enhancer additive selected from di-tertiary-butyl peroxides, 0,0-t-amyl-0 (2-ethyl hexyl) monoperoxycarbonate, 1,1 bis-(t-butylperoxy)-3,3,5-tri-methylcarbonate, tertiary butyl hydroperoxide, t-amyl perbenzoate, in an amount between approximately 1% and 15% by volume; and an additive to prevent injector clogging problems in an amount of between approximately 0.05% and 1% by volume.

It is the object of the present invention to provide a diesel fuel containing alcohol which can replace the petroleum based diesel fuel useful for a diesel engine.

It is another object of the invention to provide an alcohol based diesel fuel which excels in phase stability, autoignition and co-solvency qualities.

Yet another object of the present invention is to provide an alcohol based fuel which has significant reduction in NO_x , CO_x , CO_2 , sulphur and particulate matter in its emissions.

It is another object of the present invention is to provide an alcohol based diesel fuel which can be manufactured in a cost effective way.

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It is a further object of the present invention is to provide an alcohol based fuel which has the combustion characteristics of conventional straight diesel fuel.

It is still a further object of the present invention to provide an alcohol based fuel which should provide engine lubrication to preclude the need for expensive retrofits.

BRIEF DESCRIPTION OF THE DRAWING

FIG. 1 is a graph of the percentage brake thermal efficiency versus horsepower for volumes of ethanol in diesel.

FIG. 2 illustrates the chemical structures of the auto ignition enhancer additives I-V.

FIG. 3 is a graph of the percentage brake thermal efficiency versus horsepower for ethanol/diesel fuel blends having various autoignition enhancers included.

FIG. 4 is a graph of the percentage brake thermal efficiency versus horsepower for an ethanol/diesel blend having various amounts of additive I.

FIG. 5 is a graph of the percentage brake thermal efficiency versus horsepower for differing amounts of ethanol having various amounts of additive I and II.

FIG. 6 is a graph showing the superiority in percentage brake thermal efficiency versus horsepower for the fuel composition of the present invention.

FIG. 7 is a graph comparing the exhaust emissions of NO and NO₂ for diesel and for the fuel of the present invention.

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FIG. 8 is a graph comparing the CO emissions versus horsepower for diesel and for the fuel of the present invention.

DETAILED DESCRIPTION OF THE INVENTION

At the outset, the present invention is described in its broadest overall aspects with a more detailed description following. In its broadest overall aspect, the present invention is an alcohol based fuel composition for a diesel internal combustion engine. The composition includes additives to enhance autoignition and phase stabilizers to prevent phase separation, in specific concentrations, to produce an alcohol containing diesel fuel with almost the same performance in brake thermal efficiency as straight diesel fuel.

The fuel composition of the present invention contains approximately 20-70% by volume of a lower order alcohol, approximately 1-15% by volume of an additive to enhance autoignition and co-solvency, and approximately 4.5-5.5% by volume of a higher order alcohol to impart phase stability, with the balance being substantially diesel fuel. Each of the components that make up the fuel of the present invention were chosen based upon their performance and cost. A major concern in developing the fuel was to provide a cost effective fuel, one which could be manufactured at minimum cost and desirable fuel combustion qualities. Since all of the components used in the fuel are more expensive than the base diesel fuel, obtaining the most benefit without incurring a considerable cost was paramount.

Ethanol is the lower order alcohol in the fuel blend of the present invention, in an amount of approximately 20-70%. It was discovered that ethanol replacing diesel fuel,

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severely degraded the engine's operation without the presence of additives, as shown from tests conducted measuring brake thermal efficiency versus horsepower. Brake thermal efficiency is the work done by an engine versus the total fuel consumption, or the percentage of fuel converted into energy. It is measured by placing a load on an engine and measuring the total fuel consumption. FIG. 1 shows the effect of replacing diesel with 10%, 20%, 25% and 30% by volume ethanol, along with 100% diesel. Initially, a test was performed with 100% diesel fuel (Philips D2 Diesel Control Fuel) to find a control value for optimum brake thermal efficiency (labelled A). The brake thermal efficiency of all other compositions were compared to this optimum brake thermal efficiency for 100% diesel fuel. The lowest percentage of ethanol, 10% by volume ethanol (labelled B) performed nearly identical to the straight diesel through the load range. However, increasing the percentage of ethanol, up to 30% by volume (labelled E), severely degraded the engine's operation. 25% and 20% by volume ethanol are labelled C and D respectively. Thus, it can be seen that without the presence of additives, a 30/70 ethanol/diesel blend does not perform to the level of diesel fuel.

Any of the additives shown in FIG. 2 can be used in the composition of the present invention. Di-tertiary-butyl peroxide (additive I) is the preferred additive for use in the present invention. The selection was based on the performance of the additives, in brake thermal efficiency, versus cost. FIG. 3 shows the performance of the additives, for adding 3.85% by volume of each additive to a 50/50 ethanol/diesel blend. A 50/50 ethanol/diesel blend is most unstable in the engine without additives and so it was used as a screening blend to test the additive effectiveness. 0,0-t-amyl-0 (2-ethyl hexyl) monoperoxy carbonate (additive II), performed the best (labelled B), followed by

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di-tertiary-butyl peroxide (additive I), and tertiary butyl hydroperoxide (additive IV), having nearly equal performance characteristics (labelled C and D respectively), and then 1,1 bis-(t-butylperoxy)-3,3,5-trimethyl-cyclohexane (additive III) (labelled E), and t-amyl perbenzoate (additive V) (labelled F), with additive V performing remarkably worse than the others. 100% diesel fuel is labelled A. Di-tertiary-butyl peroxide (additive I) was chosen for the inclusion in the preferred embodiment of this invention, instead of 0,0-t-amyl-0 (2-ethyl hexyl) monoperoxycarbonate (additive II), because it is considerably cheaper than the other additives and performed sufficiently well.

A 1-2% by volume of additive in the 30/70 ethanol/diesel blend produces the best brake thermal efficiencies. With this amount of peroxide, the blend improved the thermal efficiency of the composition to almost that of the straight diesel. FIG. 4 shows the results for the addition of 0%, 1% and 2% by volume of di-tertiary-butyl peroxide (labelled D, C and B respectively) to a 30/70 ethanol/diesel blend. 100% diesel fuel is labelled A. In an important embodiment of the present invention the di-tertiary-butyl peroxide (additive I) will make up at least approximately 1% of the volume of the total fuel composition and preferably approximately 2% in the 30/70 ethanol/diesel blend.

Higher percentages of alcohol will require higher percentages of additive, in order to obtain brake thermal efficiencies comparable to straight diesel. The results for increases in the ethanol content are shown in FIG. 5. Two runs with 70% ethanol and 30% diesel were conducted. The blend with 15% additive I did very well, producing brake thermal efficiencies equal to or greater than straight diesel (labelled B). The 10% additive I blend in 60%

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ethanol (labelled C) produced acceptable results. The additive blend of 5% additive I and 5% additive II produced thermal efficiencies equal to the pure diesel fuel only at higher loads (labelled D). Similarly, the 3.85% additive I in 50% ethanol (labelled E) produced acceptable results only at high loads. 100% diesel fuel is labelled A. In an important embodiment of the present invention, the di-tertiary-butyl peroxide (additive I) will constitute up to approximately 15% in the ethanol/diesel blend for percentages of ethanol up to approximately 70%.

Butanol is the preferred higher order alcohol for the final fuel blend. As used herein "higher order alcohol" has at least 3 carbons up to about 20 or more carbons and will typically be propanol, butanol, hexanol and dodecanol. Butanol was chosen as the most cost effective surfactant. Higher order alcohols are well known as effective surfactants or surface active agents and increase phase stability and reduce separation problems associated with water-ethanol mixtures. They act as an interface between the alcohol-water solution and the diesel fuel. The fuel composition of the present invention would otherwise separate if water is present in the mixture. As is well known, water that is difficult to remove may be present in an alcohol, and/or water may be picked up by the fuel composition from the environment. To prevent this occurrence, it is desirable to add a surfactant to the fuel composition of the present invention.

Since gasohol, a blend of 90% gasoline and 10% ethanol, is a widely accepted and marketed fuel, it was chosen as a standard of comparison for the 30/70 alcohol/diesel blend. Gasohol's water tolerance was determined by gradually adding water until separation occurred. Tests found that 0.40% volume water caused separation in gasohol, so efforts were directed at making the alcohol/diesel blend achieve the same

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water tolerance performance. Tests showed that the fuel of the present invention held in solution with the addition of up to 0.375% H_2O , and separation occurred after the addition of 0.4% H_2O . Thus, the fuel of the present invention has a similar water tolerance performance to that of the commercially available gasohol.

A total alcohol content is set at approximately 30% by volume for the final blend configuration. The optimal ethanol content is approximately 25% by volume. The percentage of ethanol could be increased if it was more cost effective to do so and a cheaper means of ethanol production was available. However, this would require a higher amount of additive. The remainder of the alcohol content in the final blend was set at approximately 5% by volume of the higher order alcohol, butanol. Reducing the ethanol content from approximately 30% to 25% by volume and replacing it with approximately 5% by volume butanol imparts phase stability. In an important embodiment of the present invention, amounts from approximately 4.5% to 5.5% butanol are useful and most preferably butanol will be present in approximately 5% of the total volume.

An additive was used to solve injector clogging problems. This additive is available from The Lubrizol Corporation (29400 Lakeland Boulevard, Wickliffe, Ohio 44092-2298; Product No. LZ9520A). Testing showed that when this additive was used with alcohol/diesel blend, injection clogging problems were not experienced. In an important embodiment of the present invention, the anticlogging additive will make up approximately 0.05-0.1% of the total volume, and preferably 0.07% by volume.

The remaining volume of the fuel composition of the present invention will be a petroleum derived diesel fuel. The fuel used in the tests performed contained diesel fuel

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oil (Philips D2 Diesel Control Fuel), although any other diesel fuel can be used with the present invention. In an important embodiment of the present invention, the diesel fuel will make up approximately 66% of the total volume.

Therefore, in the most preferred embodiment of the present invention, the blend contained approximately 26.5% ethanol, 5% butanol, 66.43% diesel, 2% di-tertiary-butyl peroxide and 0.07% of a detergent additive. As used throughout this specification and claims, all percentages are by volume at room temperature unless otherwise specified. A series of tests were run showing the superior performance in brake thermal efficiency of the final fuel composition of the present invention. FIG. 6 shows the percentage brake thermal efficiency versus horsepower, for 0%, 1% and 2% by volume of the additive, di-tertiary-butyl peroxide, combined with the 30/70 ethanol/diesel blend (labelled E, D and C respectively), relative to the final fuel composition of the present invention (labelled B). The tests show that the fuel of the present composition approaches that of the brake thermal efficiency of straight diesel, confirming the superior performance of the present invention.

Emissions testing of the fuel of the present invention led to the discovery of its significant reduction in the emissions of major pollutants, for example, NO_x , CO_x , sulphur and particulate matter. A 30% alcohol mixture was selected on the basis of emission gains versus cost. A series of standard emission tests are shown in FIGS. 7 and 8 for NO_x and CO respectively, for the fuel of the present invention. The emissions gains recorded during this series were highly satisfactory. FIG. 7 shows a reduction of 11% in NO emissions with the fuel of the present invention with respect to straight diesel fuel (labelled C and A respectively). FIG. 7 also shows that the NO_2 emissions

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for the fuel of the present invention were higher at the lower loads and then decreased below that for diesel fuel at the highest load (labelled D and B respectively).

FIG. 8 shows, although CO emissions were up by 19% at start-up, the emissions were less than 25% under heavy load conditions compared with straight diesel fuel (labelled B and A respectively). In the early stages of combustion in the engine, gas temperatures are low due to the relatively small amount of fuel being combusted, limited elimination reactions take place. Consequently CO emissions are high at zero or light loads. As the load, or fuel-air ratio increases, CO emissions decrease due to higher temperatures and resulting elimination reactions with the CO that is formed.

Further tests show a 5-10% reduction of CO₂ emissions at high load with the fuel of the present invention. Removing 30% of the diesel also results in removing 30% of the sulphur-containing fuel. Additionally, results from measurements of particulate emission showed a decrease in smoke particulates for the fuel of the present invention. This is the simplest solution to reducing sulphur and particulate matter in diesel emissions. Furthermore, retaining 70% of diesel is considered sufficient to lubricate the engine to preclude the need for expensive retrofitting.

The invention may be embodied in other specific forms without departing from the spirit or essential characteristics thereof. The present embodiments are therefore to be considered in all respects as illustrative and not restrictive, the scope of the invention being indicated by the appended claims rather than by the

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foregoing description, and all changes which come within the meaning and range of equivalency of the claims are therefore intended to be embraced therein.

- What is claimed is:

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1. A phase stabilized, autoigniting, alcohol containing, fuel for the use in a diesel internal combustion engine comprising:

approximately 20-70% by volume of lower order alcohol;

approximately 30% to 80% by volume of diesel fuel;

approximately 4.5% to 5.5% by volume of a higher order alcohol surfactant;

at least approximately 1% and up to approximately 15% or more by volume of a tertiary alkyl peroxide selected from the group consisting of ditertiary alkyl peroxide, di-alkyl monoperoxycarbonate, tertiary alkyl peroxy tri-alkylcycloalkane, tertiary alkyl hydroperoxide and tertiary amyl perbenzoate; and

approximately 0.05% to 0.1% by volume of an anticlogging additive.

2. The fuel as set forth in claim 1 wherein said lower order alcohol is ethanol and said tertiary alkyl peroxide is a ditertiary alkyl peroxide.

3. The fuel as set forth in claim 2 wherein the ditertiary alkyl peroxide is ditertiary butyl peroxide.

4. The fuel as set forth in claim 1 wherein the lower alcohol comprises approximately 25% by volume of ethanol.

5. The fuel as set forth in claim 1 wherein said lower order alcohol is selected from the group consisting of ethanol and methanol.

6. The fuel as set forth in claim 1 wherein said higher order alcohol surfactant is defined by a carbon chain of three or greater, and is selected from the group consisting of propanol, butanol, hexanol or dodecanol.

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7. The fuel as set forth in claim 1 wherein said anticlogging additive is approximately 0.07% by volume.

8. The fuel as set forth in claim 1 which includes approximately 66.4% by volume of diesel fuel, approximately 26.5% by volume of ethanol, approximately 5% by volume of butanol, approximately 2% by volume of ditertiary butyl peroxide and approximately 0.07% by volume of an anticlogging additive.

9. The fuel as set forth in claim 1 which includes approximately 13.5% by volume of diesel fuel, approximately 66.4% by volume of ethanol, approximately 5% by volume of butanol, approximately 15% by volume of ditertiary butyl peroxide and approximately 0.07% by volume of an anticlogging additive.

10. A method of powering a diesel internal combustion engine comprising burning a fuel in said diesel internal combustion, which fuel comprises diesel oil and additives comprising of approximately 20-70% by volume of a lower alcohol, approximately 1-15% by volume of a tertiary alkyl peroxide, approximately 4.5% to 5.5% by volume of a higher order alcohol surfactant and approximately 0.05% to 0.1% by volume of an anticlogging additive.

11. The method of claim 10 wherein in the fuel, the lower order alcohol is ethanol and the tertiary alkyl peroxide is a ditertiary alkyl peroxide.

12. The method of claim 11 wherein in the fuel, the ditertiary alkyl peroxide is ditertiary butyl peroxide.

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13. The method of claim 10 wherein the lower order alcohol, ethanol, comprises approximately 25% by volume of the fuel.

14. The method of claim 10 wherein the lower order alcohol is selected from the group consisting of ethanol and methanol.

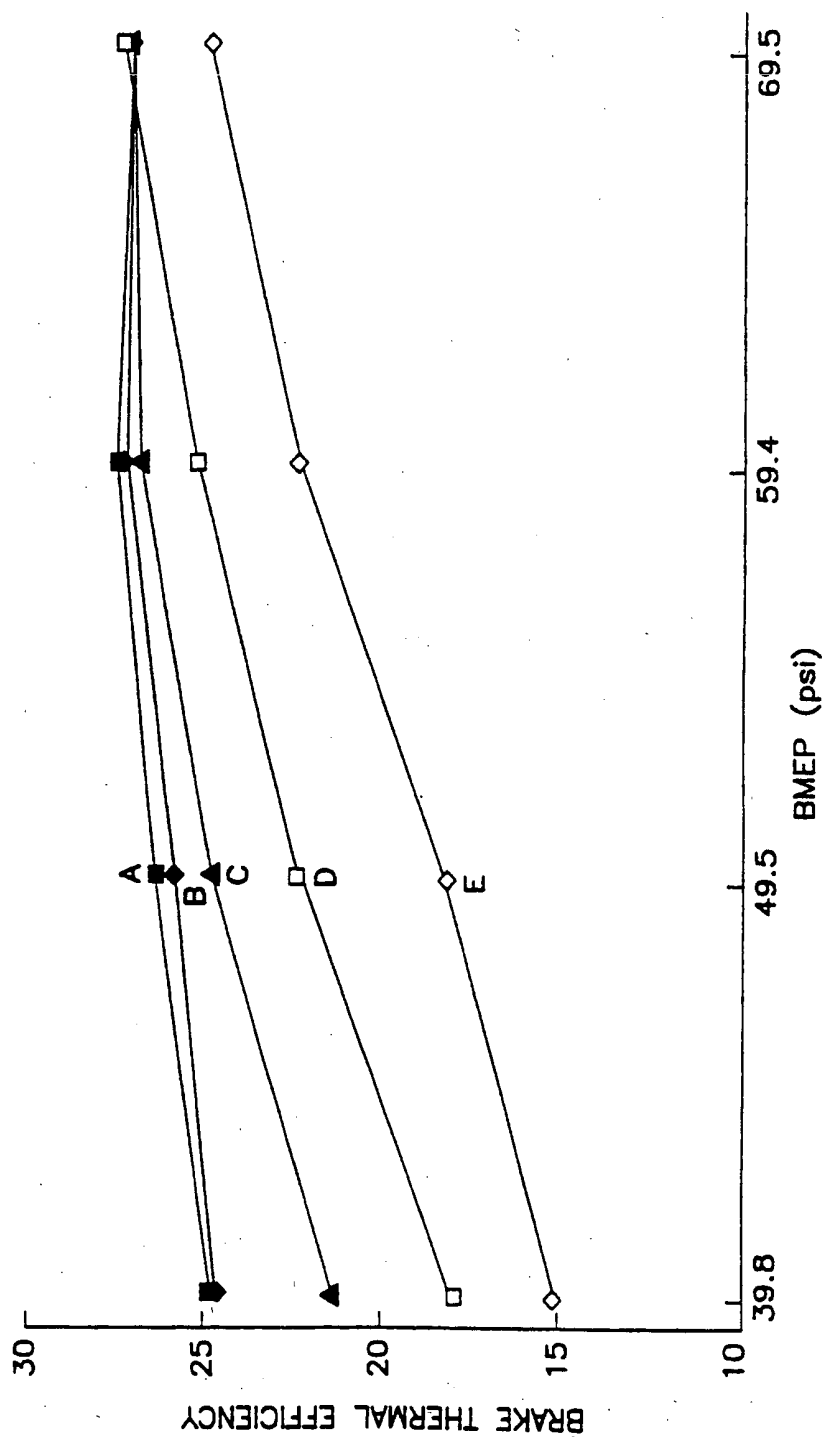
15. The method of claim 10 wherein the higher order alcohol surfactant is selected from the group consisting of propanol, butanol, hexanol or dodecanol, to reduce phase-separation problems associated with water-ethanol mixtures.

16. The method of claim 10 wherein the fuel includes approximately 66.4% by volume of diesel fuel, approximately 26.5% by volume of ethanol, approximately 5% by volume of butanol, approximately 2% of ditertiary butyl peroxide and approximately 0.07% detergent additive.

17. The method of claim 10 wherein the fuel includes approximately 13.5% by volume of diesel fuel, approximately 66.4% by volume of ethanol, approximately 5% by volume of butanol, approximately 15% of ditertiary butyl peroxide and approximately 0.07% detergent additive.

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FIG. 1



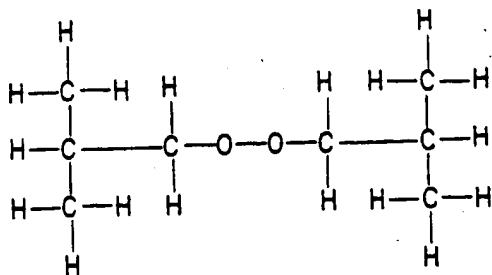


FIG. 2A

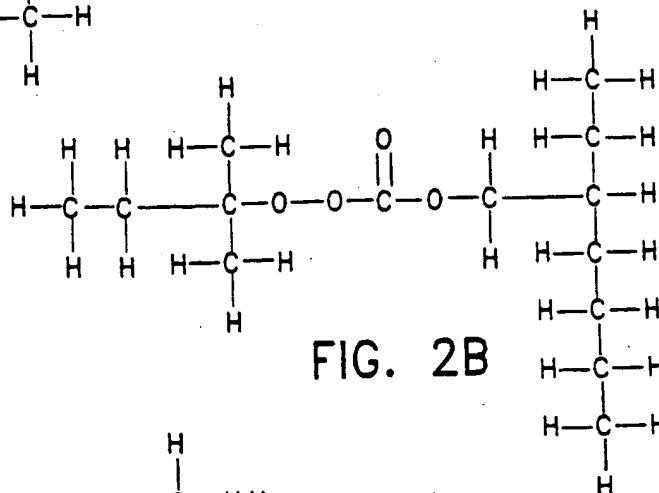


FIG. 2B

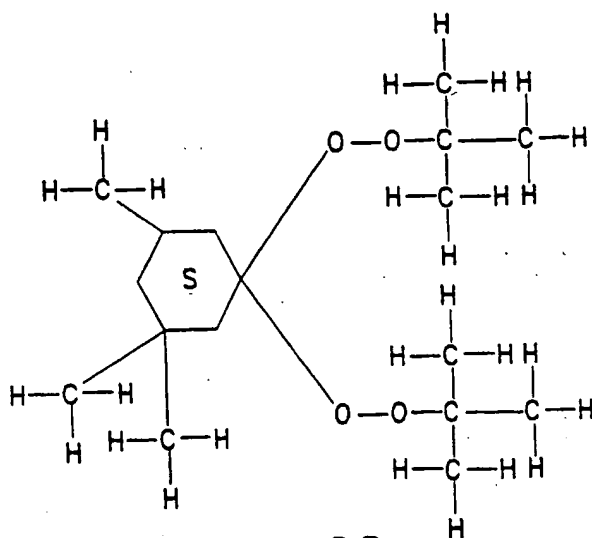


FIG. 2C

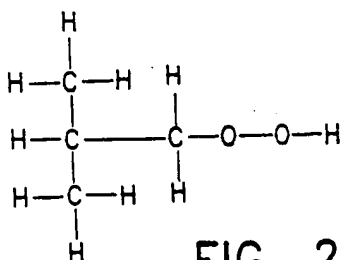


FIG. 2D

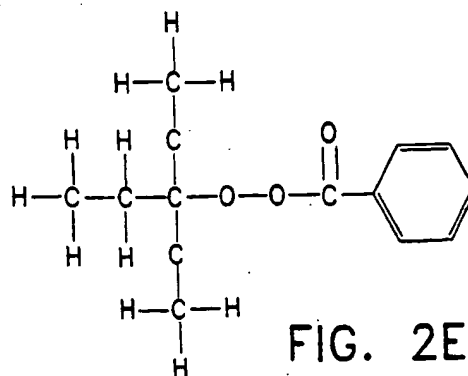


FIG. 2E

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FIG. 3

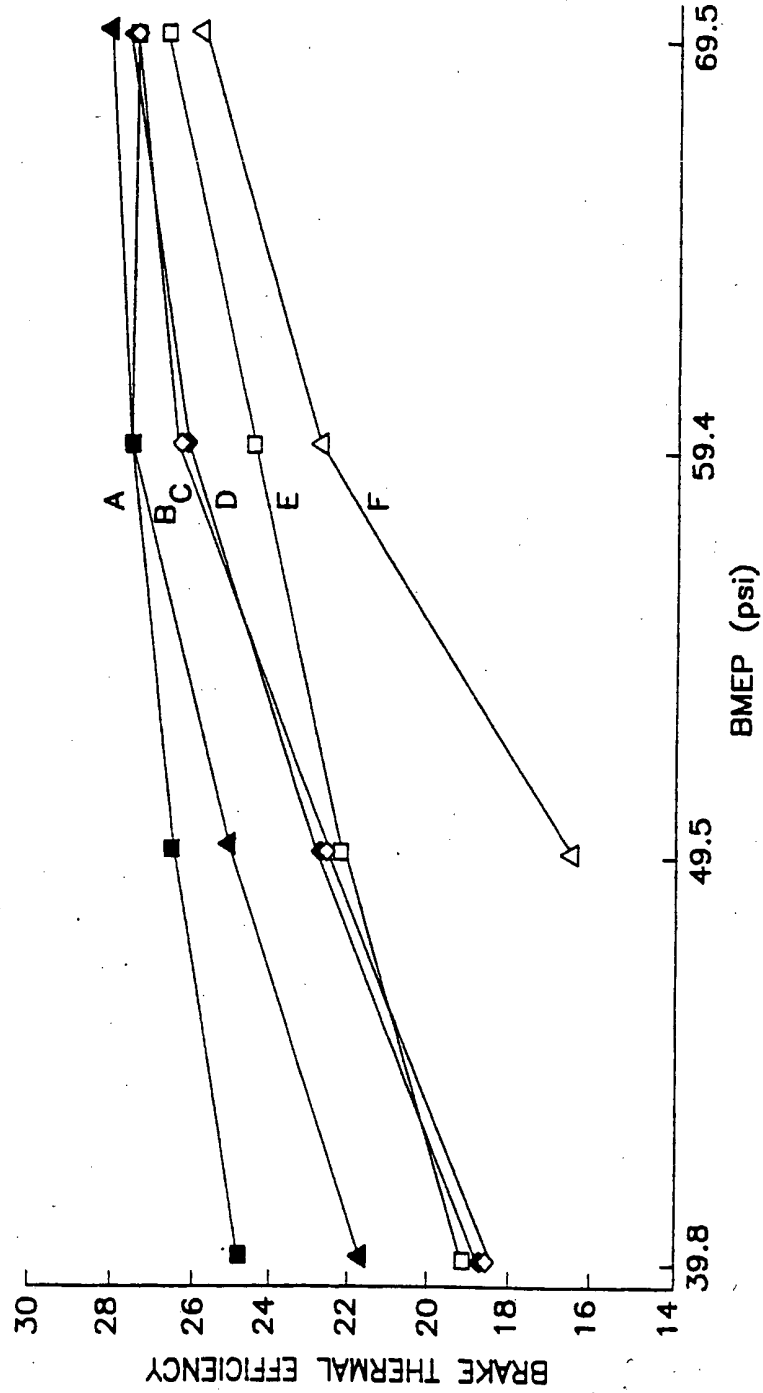
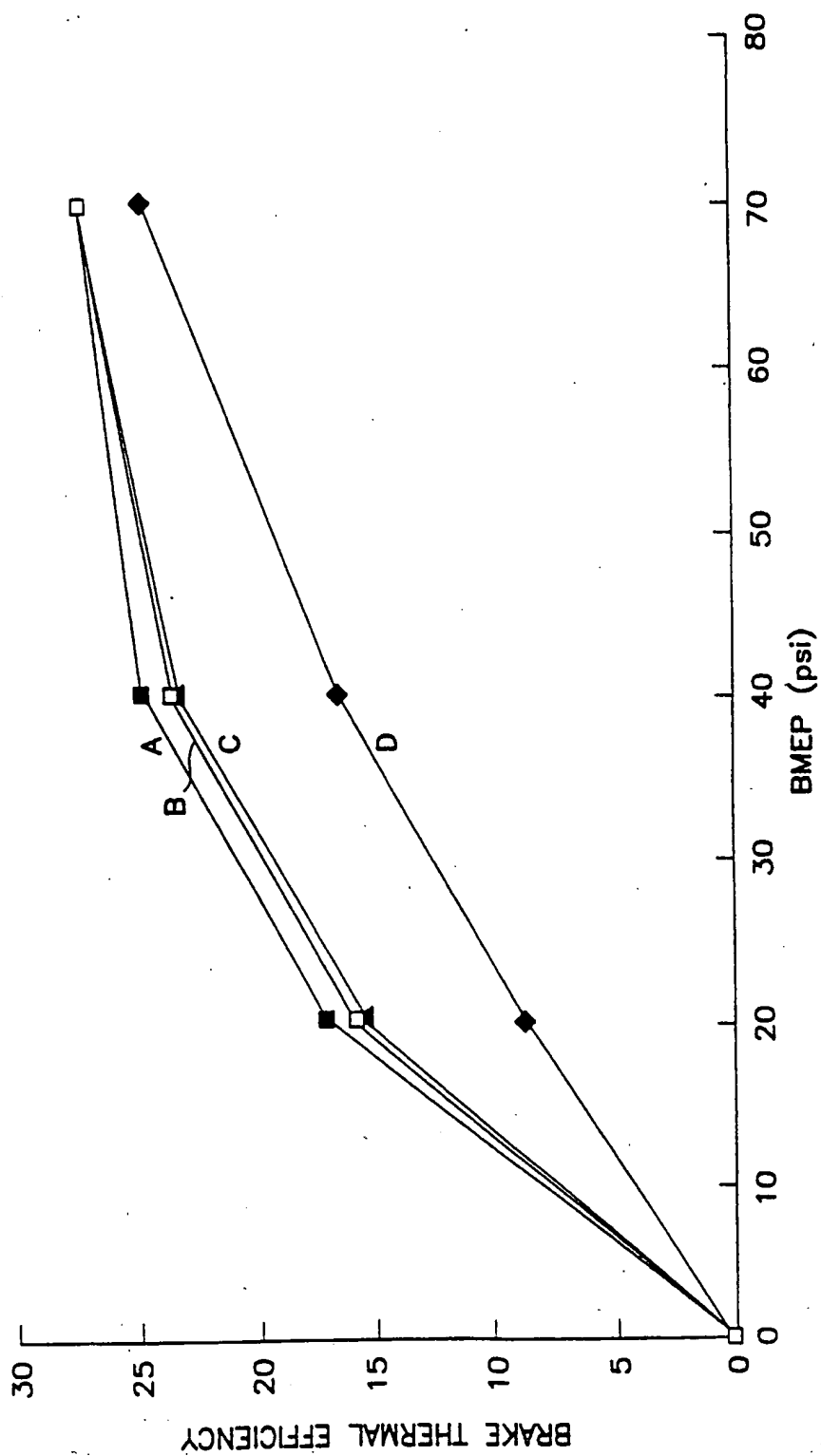


FIG. 4



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FIG. 5

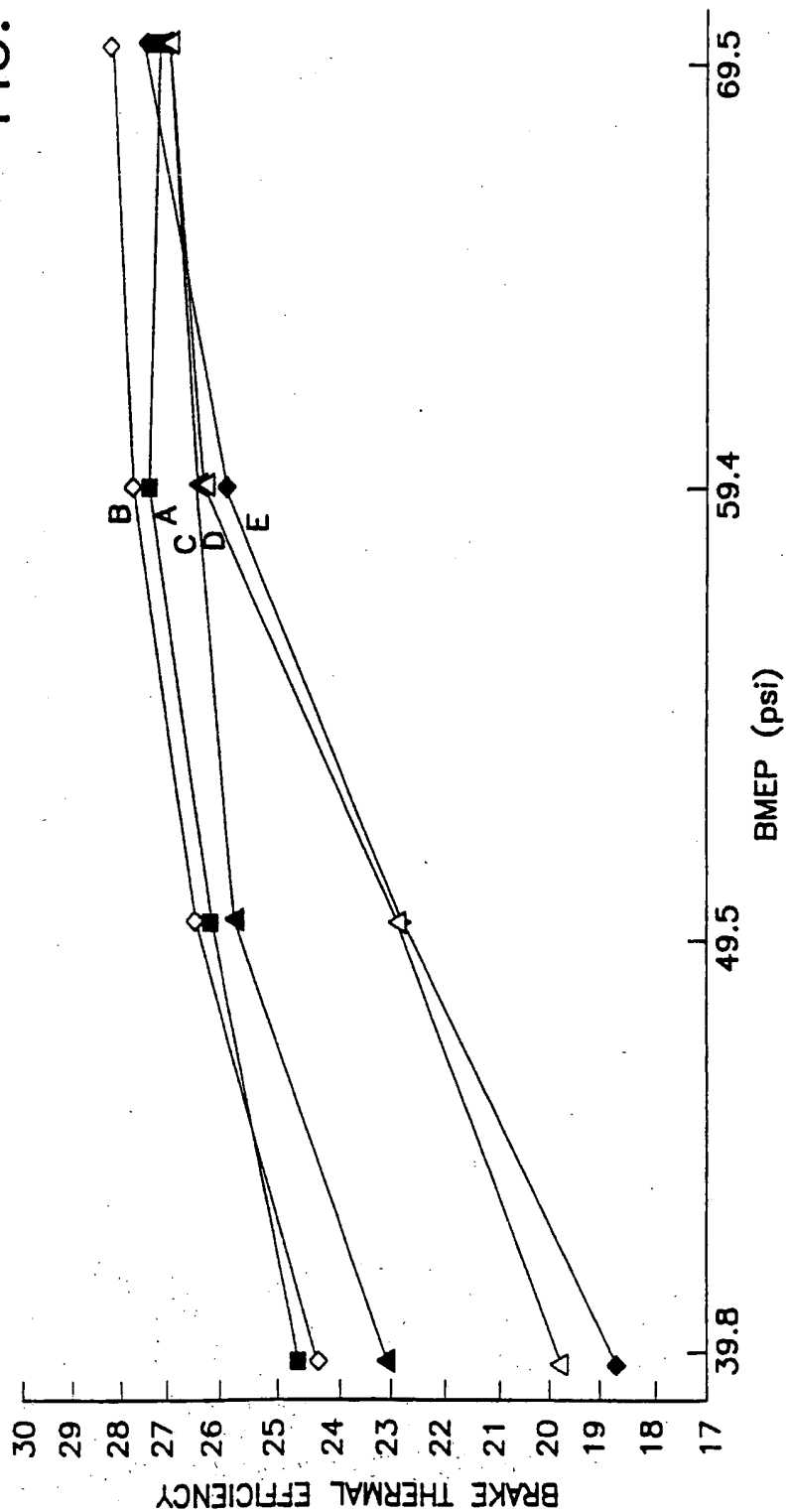
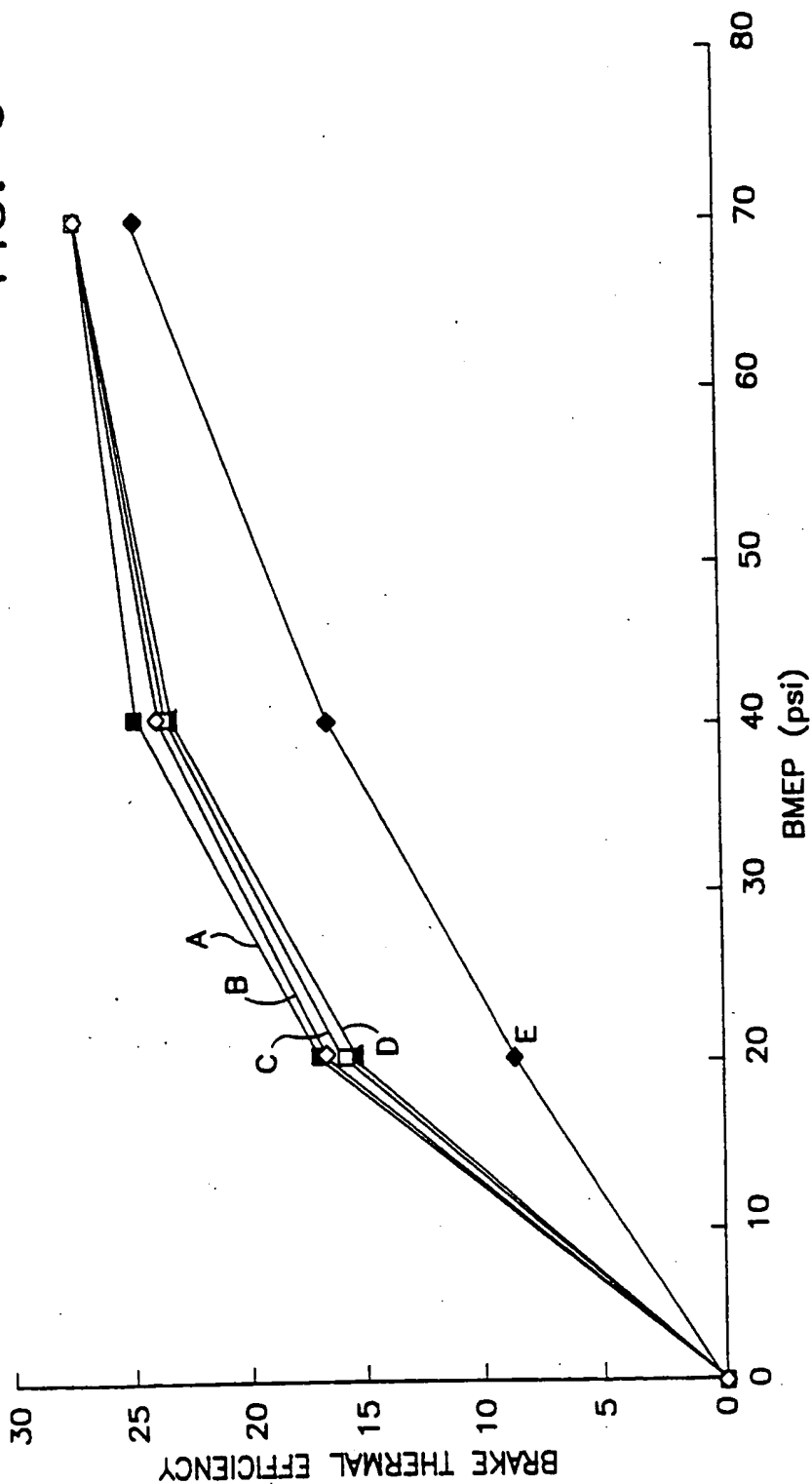


FIG. 6



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FIG. 7

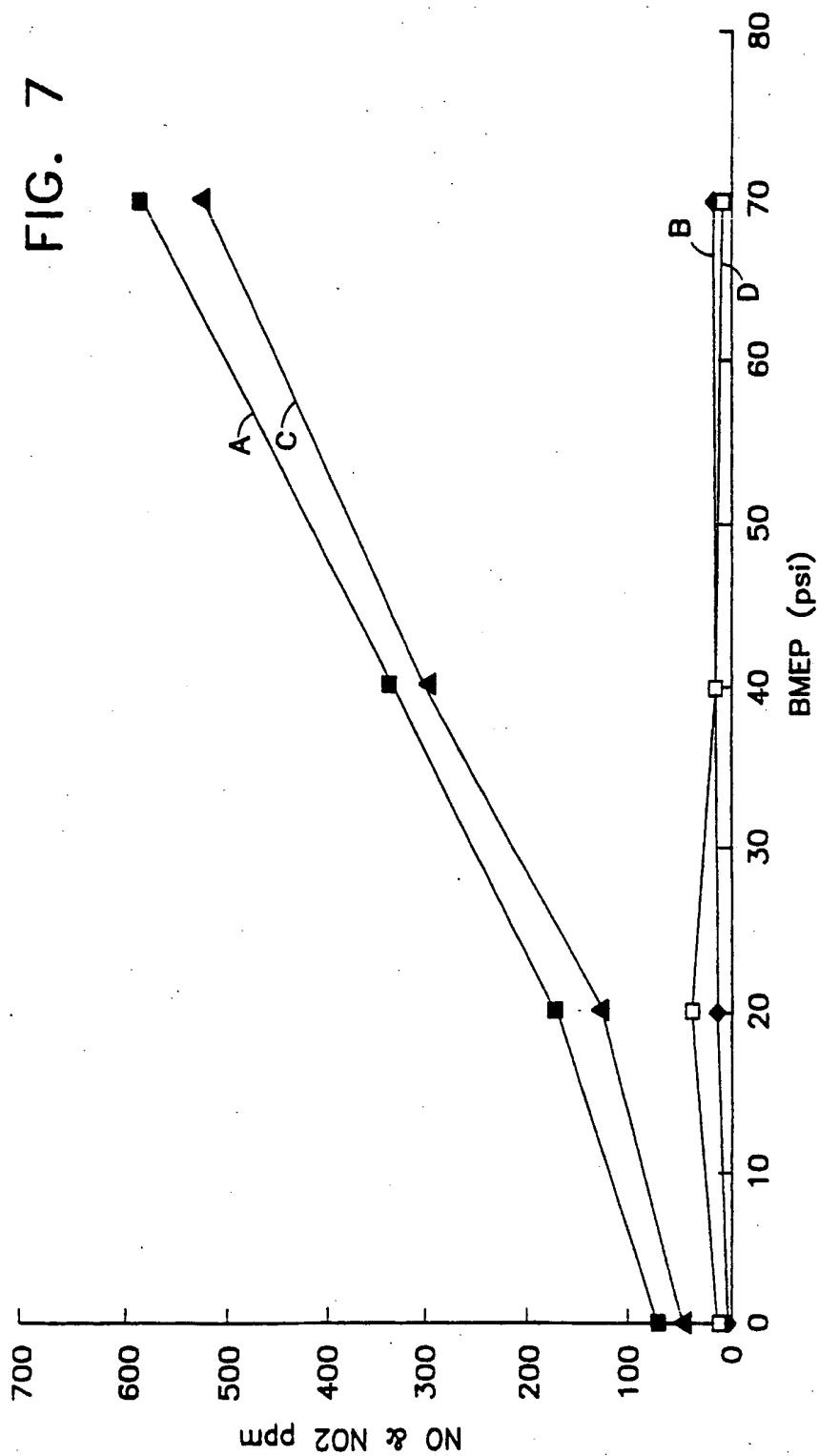
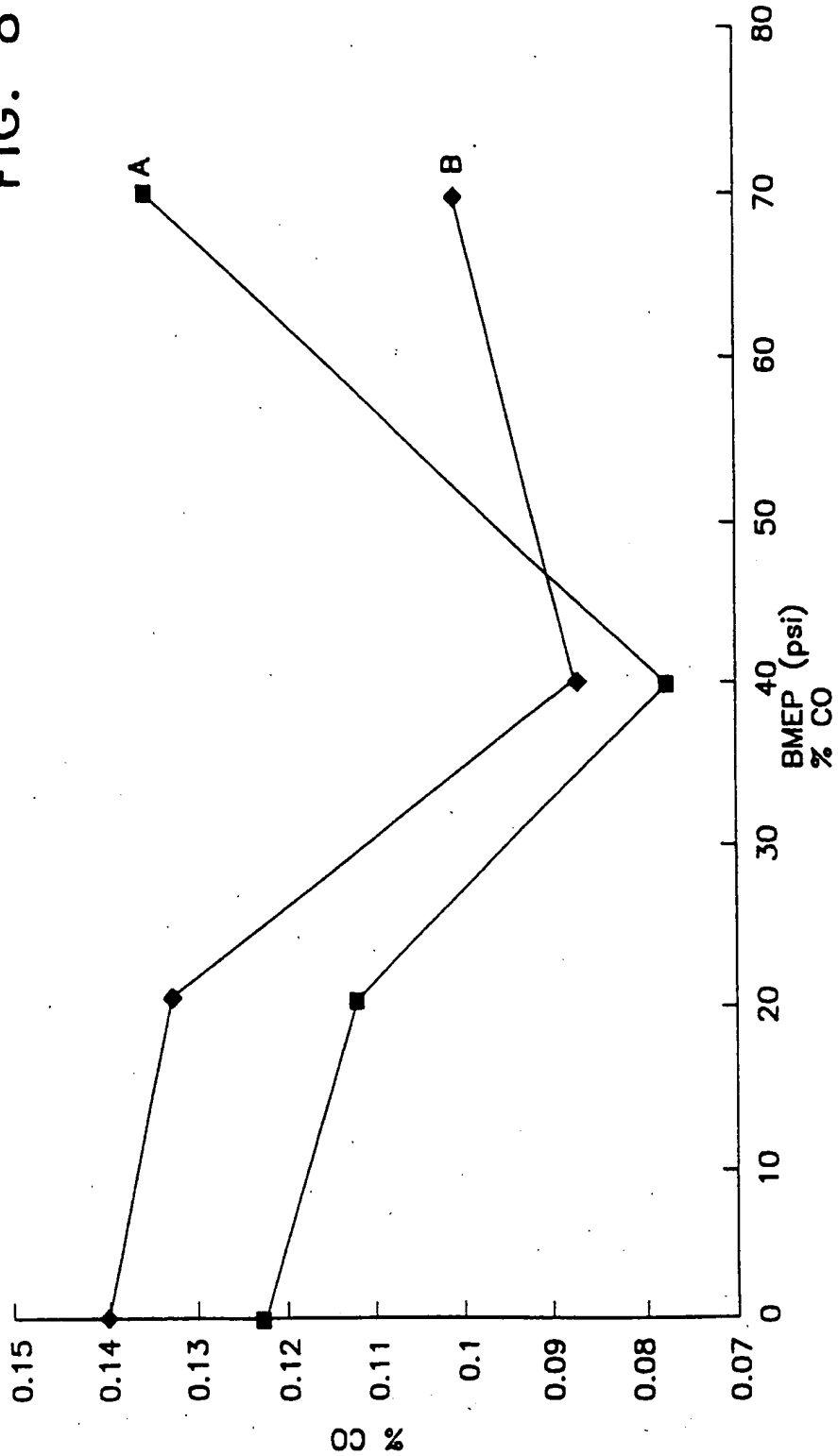


FIG. 7

FIG. 8



INTERNATIONAL SEARCH REPORT

International application No.
PCT/US93/05103

A. CLASSIFICATION OF SUBJECT MATTER

IPC(5) : C10L 1/18

US CL : 44/300, 302, 322

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 44/300, 302, 322

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched
NoneElectronic data base consulted during the international search (name of data base and, where practicable, search terms used)
None

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US,A, 2,891,851 (Bailey et al) 23 June 1959 (Col. 1, lines 50-55 and col. 2, lines 3-5).	1-3, 8-12, and 16-17
Y	US,A 4,053,428 (Pinder et al) 11 October 1977 (Col. 12, lines 24-66).	1-17
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Y	US,A, 4,509,953 (ITOW) 9 April 1985 (Col. 1, lines 20-36; Table 2 of col. 3 and Col. 4, lines 14-23).	1-6 and 8-17


☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

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Date of the actual completion of the international search
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Date of mailing of the international search report

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